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WATER-BASED SOLID FILM LUBRICANTS.(U)
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WATER-BASED SOLID FILM LUBRICANTS

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report presents the initial results of an investigation to develop high temperature 538°C (1000°F) and moderate temperature 260°C (500°F) range water-based solid film lubricants for naval aircraft applications. Lithium silicate films are shown to provide excellent water resistance compared to sodium silicate films; however, the poor permeability exhibited by lithium silicate films requires corrosion inhibition. An experimental strategy technique has been used to determine an optimum formulation for enhanced durability of this type

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of solid film lubricant. A significant increase in endurance life has been observed as a result of this approach. A limited investigation on the use of a water-dilutable organic resin bonded solid film lubricant is also reported.

SUMMARY

INTRODUCTION

A need exists for a water resistant high temperature solid film lubricant which provides corrosion protection. Specification MIL-L-81329 covers a water based solid film lubricant which contains molybdenum disulfide and graphite and uses sodium silicate as a binder. This material has excellant high temperature capabilities 538°C (1000°F), but lacks water resistance and corrosion preventive capabilities.

Specification MIL-L-8937 covers an organic solvent/resin bonded solid film lubricant with molybdenum disulfide as the major lubricating pigment. This specification was upgraded to include a sulfurous acid/salt spray corrosion test because of corrosion problems with solid film lubricant coated fasteners. Naval activities in certain geographical locations are limited to a minimum amount of organic solvents which can be emitted into the air; therefore, the development of a water based solid film lubricant would alleviate the problem of meeting these requirements.

The dual objectives of this program are to develop a high temperature water based solid film lubricant with good water resistance and corrosion protection properties and to develop a water based version of MIL-L-8937 material to eliminate the use of organic solvents. This program is being performed under AIRTASK A320320A/001B/1F61542000, Work Unit No. ZM501.

RESULTS

1. Lithium silicate bonded solid film lubricants have been shown to provide superior water resistance properties compared to sodium silicate; however, corrosion inhibition is still required.

2. Formulations based on optimizing the durability of lithium and sodium silicate bonded films have been uncovered based on a new experimental strategy technique.

3. A water based organic resin bonded solid film lubricant was found to also require corrosion inhibition. The durability of this lubricant was only approximately one-half that of the MIL-L-8937.

FUTURE PLANS

1. Complete studies to eliminate graphite and/or add corrosion inhibitors.

2. Prepare specification requirements for high temperature water based solid film lubricant.

3. Complete development of moderate temperature range water based organic resin solid film lubricant.

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BACKGROUND

The approach used to increase water resistance of sodium silicate bonded high temperature solid film lubricants centers on the substitution of the sodium silicate binder with lithium silicate. In recent years, it has become commercially possible to produce lithium silicate solutions, although these are not made by the usual method for the manufacture of alkali metal silicate solutions (1). This is due to the fact that fusion of lithium carbonate with silica results in lithium silicate glasses which, unlike the analogous sodium compounds, are very insoluble in water. It is precisely this property; that is, low solubility in water which should be beneficial in solid lubricant formulations. The low solubility of lithium silicate glasses is attributed to the small radius of the lithium ion and its resultant high charge density. Consequently, it is highly hydrated and the size of the hydrated ion is actually greater than that of other common alkali metal ions. In addition to the alternate binder study, a substitute for graphite and corrosion inhibitors which are compatible with lithium silicate will be investigated.

Water based solid film lubricants for moderate temperature range applications will be developed utilizing water soluble organic resins such as acrylics. Water soluble corrosion inhibitors will be required to meet the corrosion preventive requirements of MIL-L-8937.

RESULTS AND DISCUSSION

HIGH TEMPERATURE RANGE WATER BASED SOLID FILM LUBRICANT

Endurance Life

Table 1 lists the composition and properties of various silicate solutions investigated in this program. Sodium silicate is used to prepare MIL-L-81329 solid film lubricants and is included for comparison purposes. Table 2 shows formulation data for the preparation of solid lubricant coatings. In initial studies, all formulations were prepared with total solids content identical to the MIL-L-81329 (sodium silicate) material. For example, from Table 2 for the sodium silicate formulation, 50g of silicate solution is called for. From Table 1, the total solids contained in 50g of sodium silicate is .428 x 50g which represents 21.4g. In order to maintain this amount of solids in the other formulations, it was necessary to determine what quantity of solution contained 21.4g of solids. In the case of lithium silicate A, this solution contained 22.9% solids; therefore, it was necessary to use 94g of solution. Also, the amount of water was adjusted to compensate for differences in total solids content. The films produced by the lithium silicate solutions were identical to those prepared with sodium silicate in appearance, texture, sprayability and adhesion.

Initial testing centered on determing the water resistance properties of the resultant films. Solid lubricant coated anodized aluminum panels were prepared by spray application to a thickness of 0.0127 mm (0.0005 inch) and cured in accordance with paragraph 5.1.1 of MIL-L-81329.

Figure 1 shows the results obtained for three panels immersed in water under different conditions. The panel on the left, which is coated with MIL-L-81329 (sodium silicate), was immersed in distilled water at ambient temperature for five minutes. Removal of the film by rubbing is evident although a slight coating remains which resembles burnished MoS_2. An identical panel (Figure 1, middle) immersed in boiling water for only 10 seconds resulted in complete removal of the film exposing the shiny aluminum surface. The panel on the right, coated with lithium silicate A solid film lubricant, was immersed in boiling water for eight hours with no deterioration of the film. These results demonstrate the superior water resistance properties which can be achieved with the use of lithium silicate as a binder.

Having established that a superior water resistant film could be achieved, the next question to be considered was re the new lithium silicate bonded solid film lubricants as good as sodium silicate bonded films in terms of lubricating ability? The answer to this question was obtained in the next phase of this program in which Falex lubricant endurance life tests were performed in accordance with reference (2), except that the test specimens were grit blasted with 120 steel grit to produce a surface finish of 50-60 RMS. Figure 2 shows a schematic diagram of the Falex test configuration. The V-blocks are loaded against the pin which is rotated at 290 RPM. The number of minutes to failure, which is indicated by a 1152 g-cm (10 lb-in) torque rise above the steady state, is the endurance life of the lubricant.

Table 3 shows the results of two series of tests, one using AISI 3135 steel pins vs. AISI 1137 steel V-blocks, and the other substituting TZM molybdenum alloy pins for the steel pins. The TZM pins were run uncoated without surface pretreatment against the coated V-blocks. With steel pins, both pins and blocks were coated. Most of the Falex evaluations to-date were performed on lithium silicate A comparing these results to a MIL-L-81329 sodium silicate. Only limited tests have been performed on the lithium silicate B and lithium-sodium silicate. The results shown in Table 3 clearly indicate the quality of lithium silicate bonded films vs. sodium silicate bonded films in terms of lubricating ability.

Experimental Techniques

During the course of this investigation, an experimental strategy (3) was introduced which would better define composition ranges for obtaining an optimum endurance life for the solid film lubricant coating system. Using a three component approach, i.e., MoS_2 graphite and silicate binder, various formulations were prepared based on keeping the sum of the weight of components constant. A triangular graph is suitable for defining compositions of the three components. The results of this approach are shown in Table 4 and Figure 3 for sodium silicate bonded solid film lubricants. Since it is not realistic to test compositions at the apices of the triangle, i.e., where only one component is present, the following arbitrary limits on composition ranges were set. MoS_2 varied from 20 to 80%, graphite from 0 to 60% and sodium silicate from 20 to 80%. By defining these limits, a new triangular

range within the overall compositional triangle was established as shown in Figure 3. The strategy of this experimental technique involves testing compositions represented on the triangle at the apices (compositions 1, 2 and 3), at the mid-points on the lines forming the triangle (compositions 4, 5, 6), a center point (composition 7), and so called check points (compositions 8, 9, and 10). It can be observed from Table 4 and Figure 3 that an optimum endurance life is obtained at composition 8 (68 minutes). Composition 11, shown in Figure 3, represents the composition initially used in this study to compare sodium and lithium silicated bonded films. It is equivalent to 55% MoS_2, 5.5% graphite and 39.5% sodium silicate.

In a similar fashion tests were conducted using lithium silicate-A, only a smaller concentration range was studied. The concentrations ranged from 29.5% to 69.5% for MoS_2, 1.0 to 41.0% for graphite and 29.5 to 69.5% lithium silicate A. The results of this study are shown in Table 5 and Figure 4. The optimum endurance life was found at composition 7 (187 minutes). The composition of lithium silicate A initially tested is represented by composition 11 (Figure 4). This is equivalent to 40.9% MoS_2, 4.1% graphite and 55.9% lithium silicate A.

The application of this experimental strategy has resulted in obtaining optimum compositions for endurance life with a minimal amount of testing.

Sulfurous Acid/Salt Spray Corrosion

The next phase of this program centered on studying the corrosion protection properties of the silicate bonded films. It is known that MIL-L-81329 films last only one or two cycles in the sulfurous acid/salt spray corrosion test (Federal Test Method No. 791B Method 5331) before corrosion is observed. One cycle is defined as two hours exposure to the spray plus a minimum two hour dry time. MIL-L-8937 and MIL-L-23398 films are required to remain corrosion free for at least four cycles. As shown in Figure 5 steel discs (AISI 1020) coated with composition 11 for sodium silicate and lithium silicate A fail in two cycles or less. With lithium silicate A corrosion is observed after the first cycle indicating poorer corrosion resistance than sodium silicate films. Apparently films formed by lithium silicate A, although resistant to dissolution by water, are more permeable than sodium silicate films. In order to determine if the composition of the lithium silicate film could effect corrosion protection properties, compositions 1, 2, 3 and 7 from Table 5 were tested. Figure 6 shows the results of this investigation. After the first cycle compositions 2 and 3 were severely corroded. Slight corrosion was found on composition 1 while some initial pitting was observed on composition 7. Running compositions 1 and 7 for another cycle brought on severe corrosion. Inhibitors will thus be required for lithium silicate films.

MODERATE TEMPERATURE RANGE WATER-BASED SOLID FILM LUBRICANT

An initial investigation on this phase of the program was conducted on a sample of material obtained from a commercial source. This material

is described as a dispersion of solid lubricants in an air-curing, water dilutable, organic resin with an operating temperature range of -70°C (-94°F) to +250°C (482°F). In the preparation of steel specimens for endurance life testing, it was noted that the specimens started to corrode when the solid film was applied. Careful preparation of specimens was required in order to eliminate this initial action of the solid film lubricant on the test specimens. Falex endurance testing of this formulation resulted in an average wear life of 120 minutes. MIL-L-8937 films are required to run at least 250 minutes. A corrosion inhibitor for this formulation will also be required.

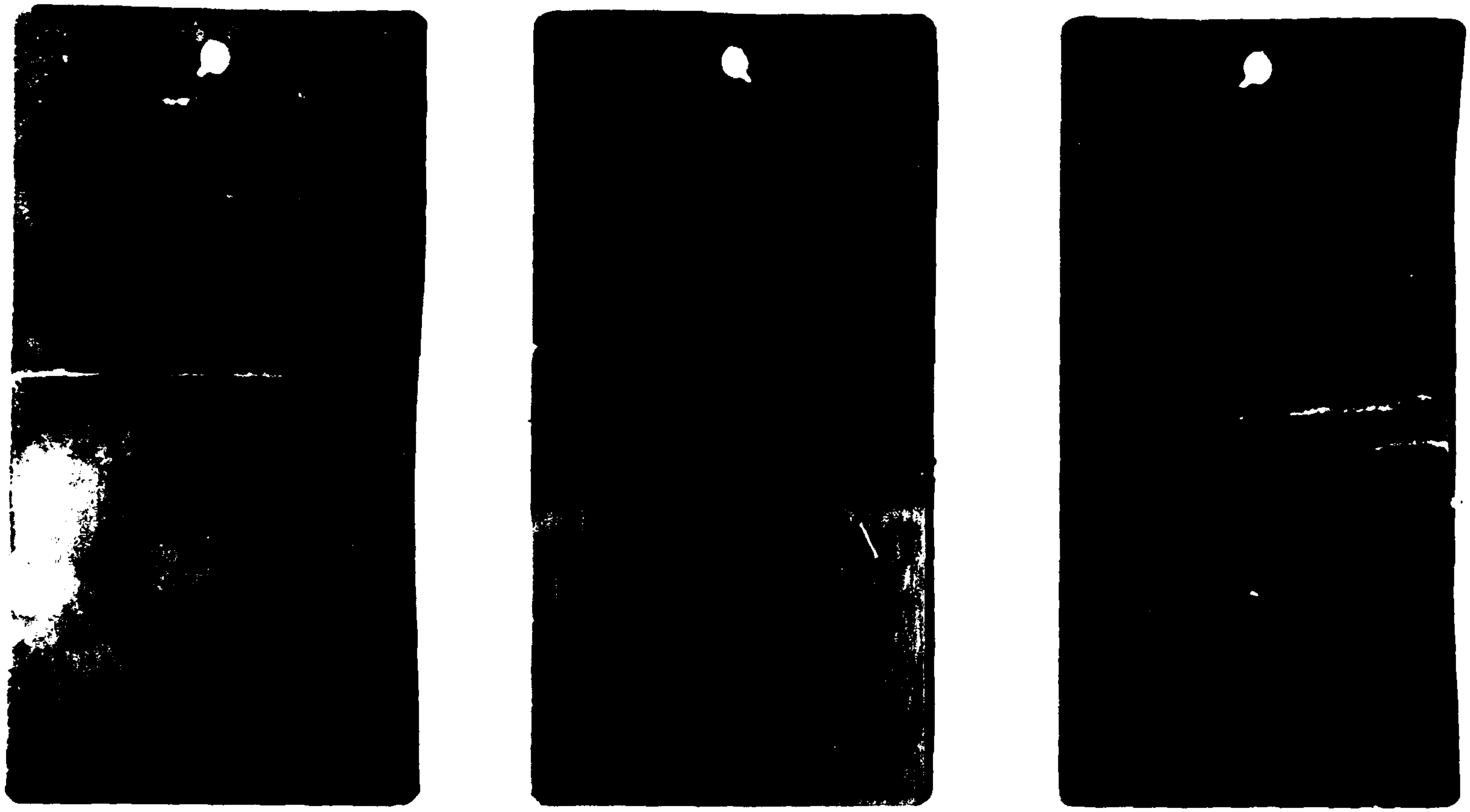
R E F E R E N C E S

(1) Sams, R. H., U. S. Patent 3,565,675, 23 Feb 1971

(2) ASTM Method D2625 Procedure A

(3) Marquardt, D. W. and Snee, "Test Statistics for Mixture Models", Technometrics, 16 pp. 533-537 (1974b)

WATER RESISTANCE OF VARIOUS SOLID FILM LUBRICANTS



MIL-L-81329 SODIUM SILICATE BINDER	MIL-L-81329 SODIUM SILICATE BINDER	LITHIUM SILICATE BINDER
H_2O, AMB. TEMP 5 MIN	H_2O, BOILING, 10 SEC	H_2O, BOILING, 8 HOURS

FIGURE 1. WATER RESISTANCE OF VARIOUS SOLID FILM LUBRICANTS

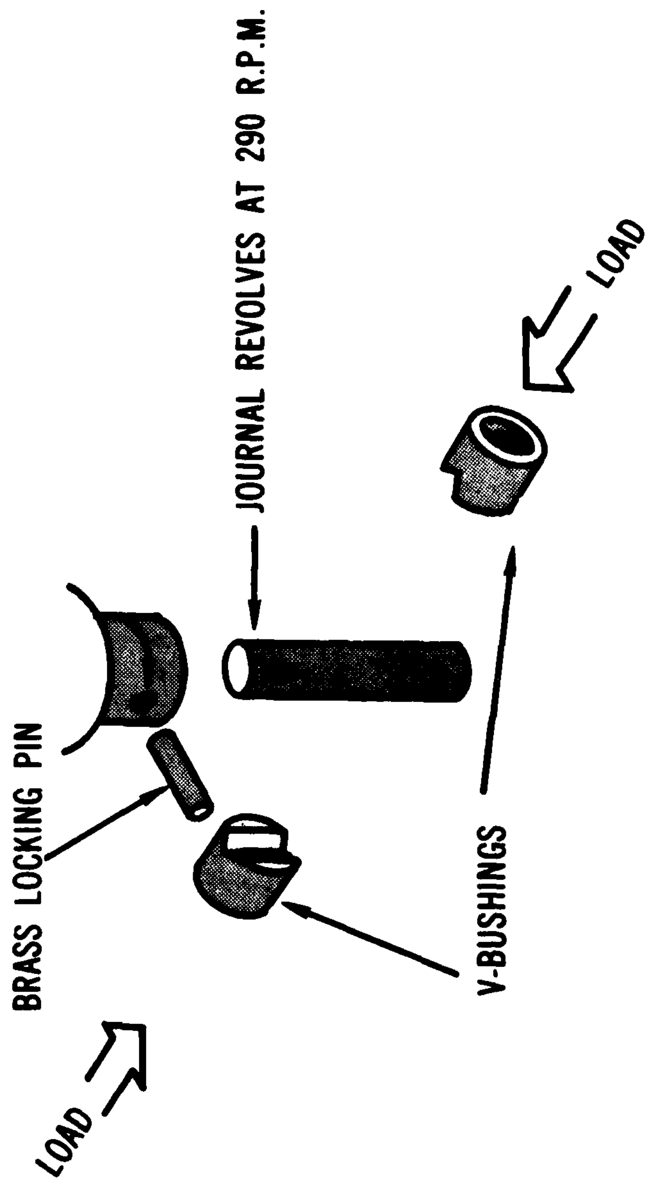


Fig. 2 FALEX TEST SPECIMEN CONFIGURATION.

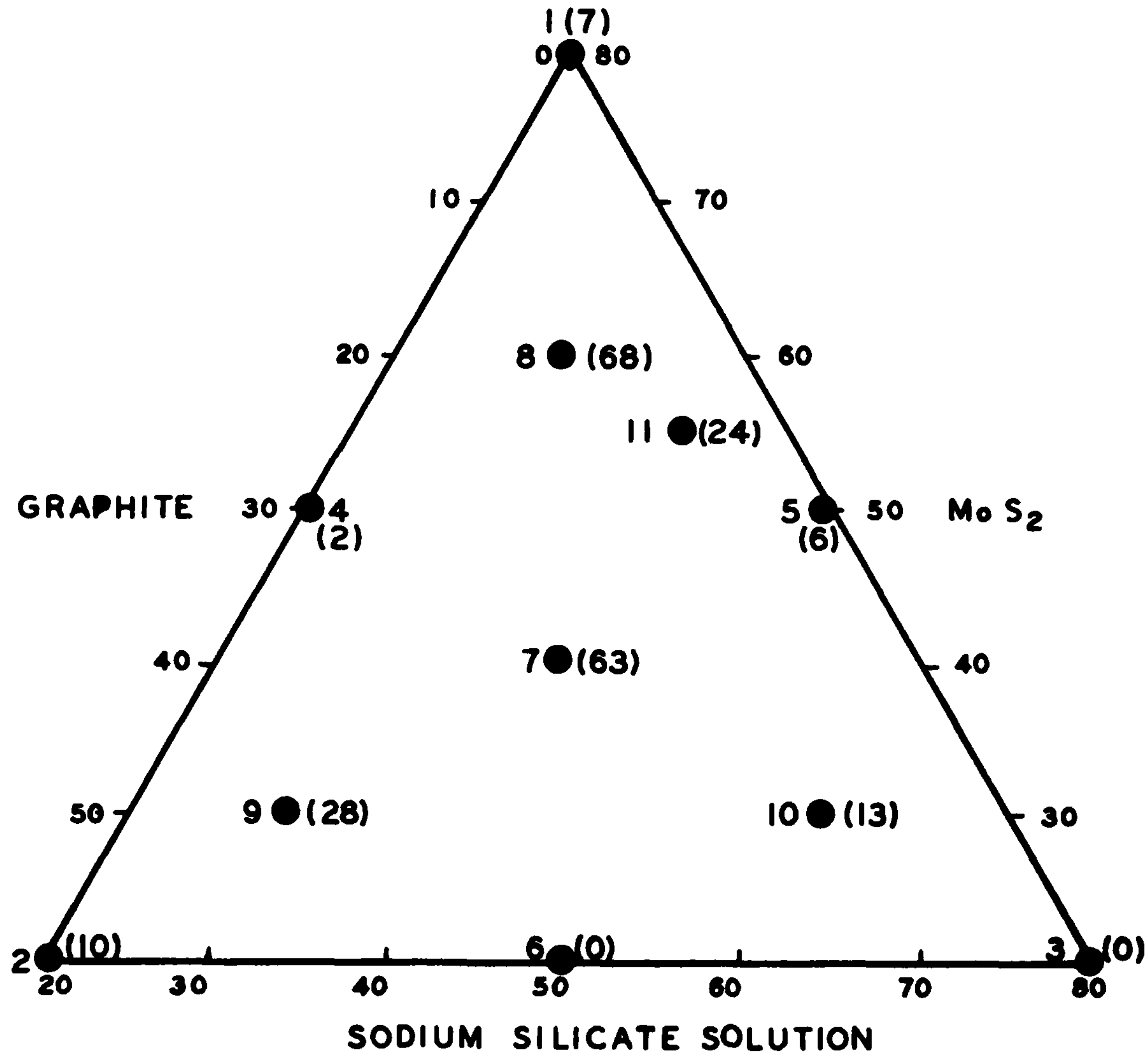


FIGURE 3. THREE COMPONENT COMPOSITION DIAGRAM FOR SODIUM SILICATE BONDED SOLID FILM LUBRICANTS

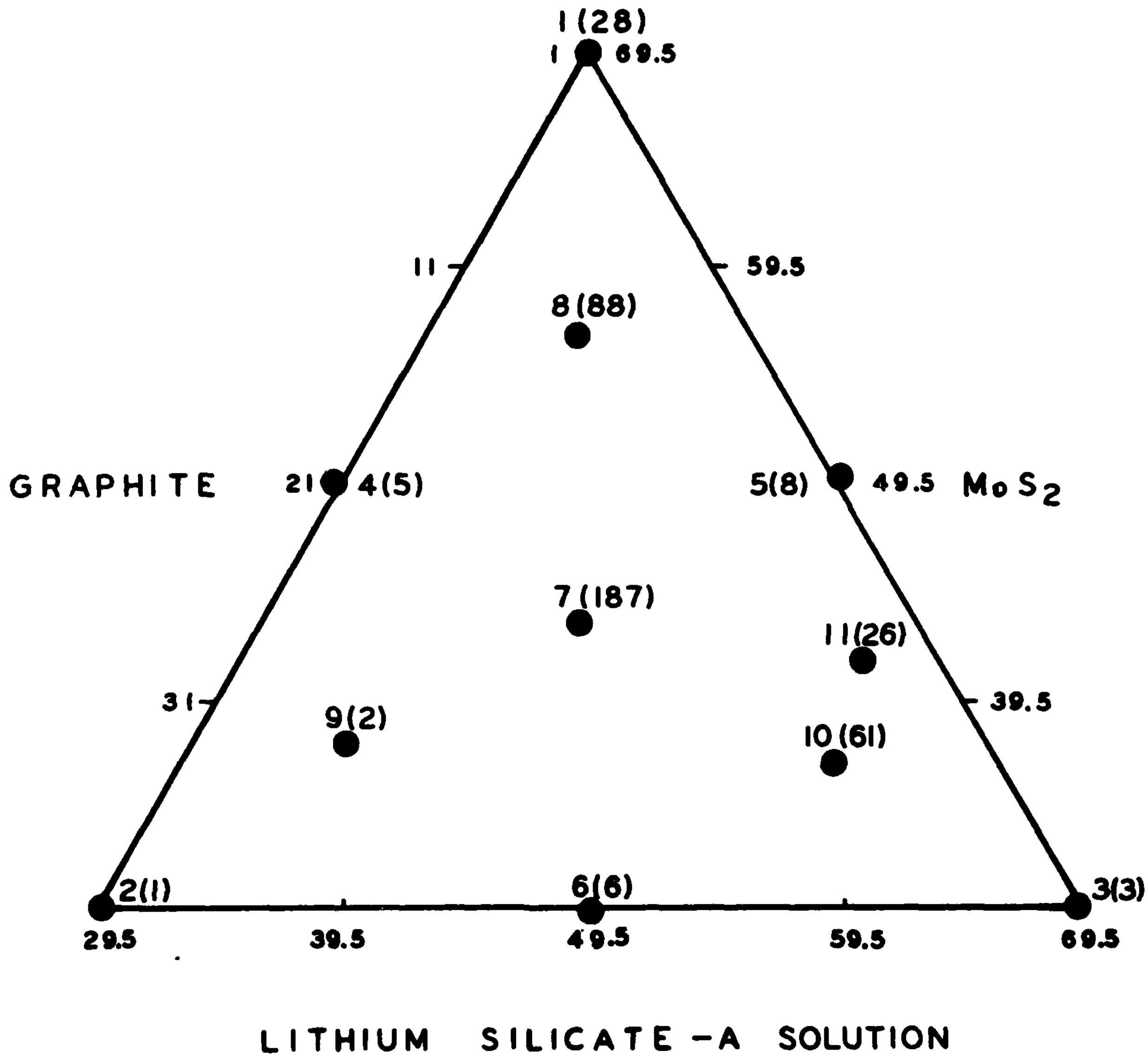


FIGURE 4. THREE COMPONENT COMPOSITION DIAGRAM FOR LITHIUM SILICATE BONDED SOLID FILM LUBRICANTS

SULFUROUS ACID/SALT SPRAY CORROSION TEST

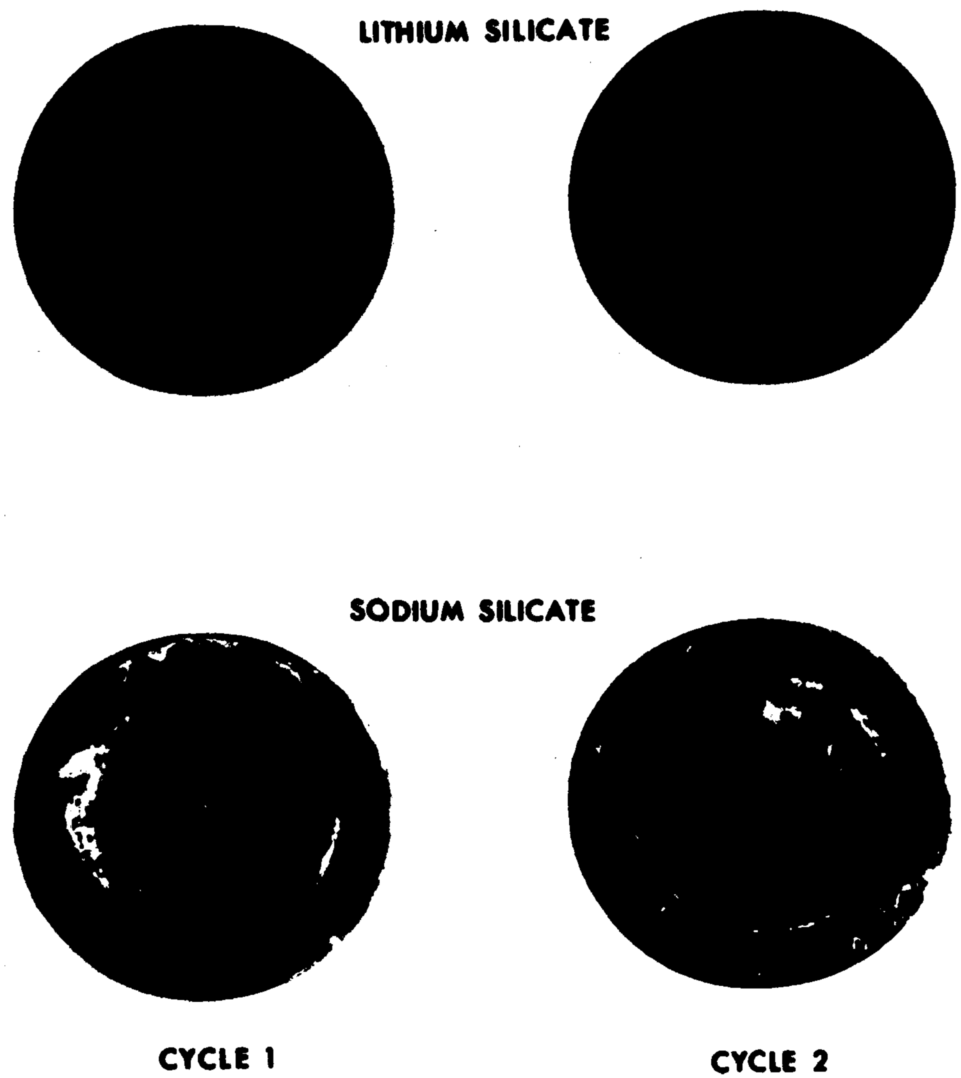


FIGURE 5. SULFUROUS ACID/SALT SPRAY CORROSION TESTS ON SODIUM AND LITHIUM SILICATE BONDED SOLID FILM LUBRICANTS

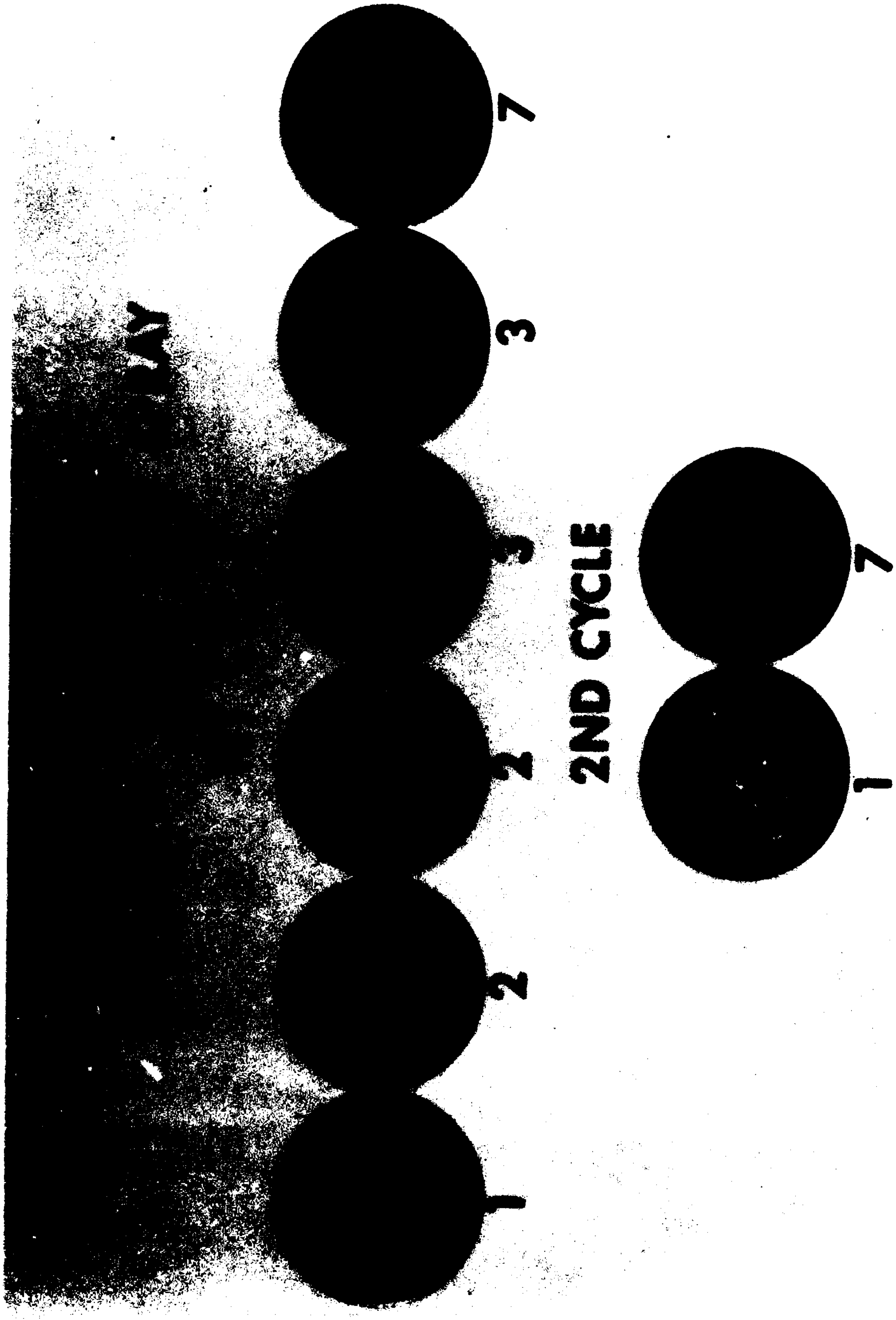


FIGURE 6. SULFUROUS ACID/SALT SPRAY CORROSION TESTS ON LITHIUM SILICATE BONDED SOLID FILM LUBRICANTS

TABLE 1. COMPOSITION AND PROPERTIES OF ALKALI METAL SILICATES

	Lithium Silicate A	Lithium Silicate B	Lithium/ Sodium Silicate	Sodium Silicate (MIL-L-81329)
Li_2O, %	2.2	1.6	1.8	----
Na_2O, %	---	---	1.2	10.9
SiO_2, %	20.7	18.8	19.6	31.9
Total Solids %	22.9	20.4	22.6	42.8
Molar Ratio:				
SiO_2/Li_2O	4.6:1	5.9:1	---	----
SiO_2/Na_2O	---	---	---	3.0:1
$SiO_2/Li_2O/Na_2O$	---	---	5.4:1:0.3	----
pH	10.7	10.7	10.7	11.5
Density, 25°C (77°F) g/cc	1.17	1.18	1.19	1.49
Viscosity, 30°C (86°F) cps	22.5	3.5	4.6	4.8

TABLE 2. FORMULATION DATA ON SILICATED BONDED SOLID FILM LUBRICANTS

Component	Lithium Silicate A	Lithium Silicate B	Lithium/ Sodium Silicate	Sodium Silicate (MIL-L-81329)
MoS_2, g	70	70	70	70
Graphite, g	7	7	7	7
Silicate Solution, g	94	105	95	50
H_2O,g	16	5	15	60

TABLE 3. FALEX TEST RESULTS, MINUTES TO FAILURE

LOAD:	4488N (1000 lb) gage load
SPEED:	290 RPM
TEMPERATURE:	Ambient
SPECIMENS:	As indicated
SURFACE PRETREATMENT:	Grit blasted with 120 steel grit

	Lithium Silicate A		Lithium Silicate B		Lithium Sodium Silicate		Sodium Silicate (MIL-L-81329)	
Falex Machine No.	671	710	671	710	671	710	671	710
Steel on Steel	24	28	21	43	41	32	23	16
(Grit Blasted)	24	28			31	31	24	31
	25	25						
	27	23						
AVG.	25	26	21	43	36	32	24	24
TZM vs. Steel	69	64					87	69
(Grit Blasted)	77	57					85	78
	84	86						
	73	73						
	63	57						
AVG.	73	67					86	74

TABLE 4. COMPOSITIONS OF SODIUM SILICATE BONDED SOLID FILM LUBRICANTS AND FALEX ENDURANCE LIFE

Composition No.	MoS_2 %	Graphite %	Sodium Silicate %	Falex Endurance Life, Min. Run 1	Run 2	AVG
1	80	0	20	5	8	7
2	20	60	20	16	3	10
3	20	0	80	0	0	0
4	50	30	20	3	1	2
5	50	0	50	7	5	6
6	20	30	50	0	0	0
7	40	20	40	59	67	63
8	60	10	30	73	63	68
9	30	40	30	33	22	28
10	30	10	60	17	8	13

Composition Limits

MoS_2 = 20 to 80%

Graphite = 0 to 60%

Sodium Silicate = 20 to 80%

TABLE 5. COMPOSITIONS OF LITHIUM SILICATE BONDED SOLID FILM LUBRICANTS AND FALEX ENDURANCE LIFE

Composition No.	MoS_2 %	Graphite %	Lithium Silicate A %	Falex Endurance Life, Min. Run 1	Run 2	AVG
1	69.5	1.0	29.5	33	22	28
2	29.5	41.0	29.5	1	1	1
3	29.5	1.0	69.5	1	5	3
4	49.5	21.0	29.5	6	4	5
5	49.5	1.0	49.5	9	6	8
6	29.5	21.0	49.5	6	5	6
7	42.8	14.4	42.8	235	139	187
8	56.2	7.7	36.2	84	91	88
9	36.2	26.6	36.2	2	2	2
10	36.2	7.7	56.2	65	56	61

Composition Limits

MoS_2 = 29.5 to 69.5%

Graphite = 1 to 41%

Lithium Silicate A = 29.5 to 69.5%

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